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S. W. STRATTON, DIRECTOR

No. 157

AN INVESTIGATION OF THE PHYSICAL PROPERTIES OF DENTAL MATERIALS

BY

WILMER H. SOUDER, Associate Physicist
CHAUNCEY G. PETERS, Associate Physicist

Bureau of Standards

ISSUED MAY 22, 1920



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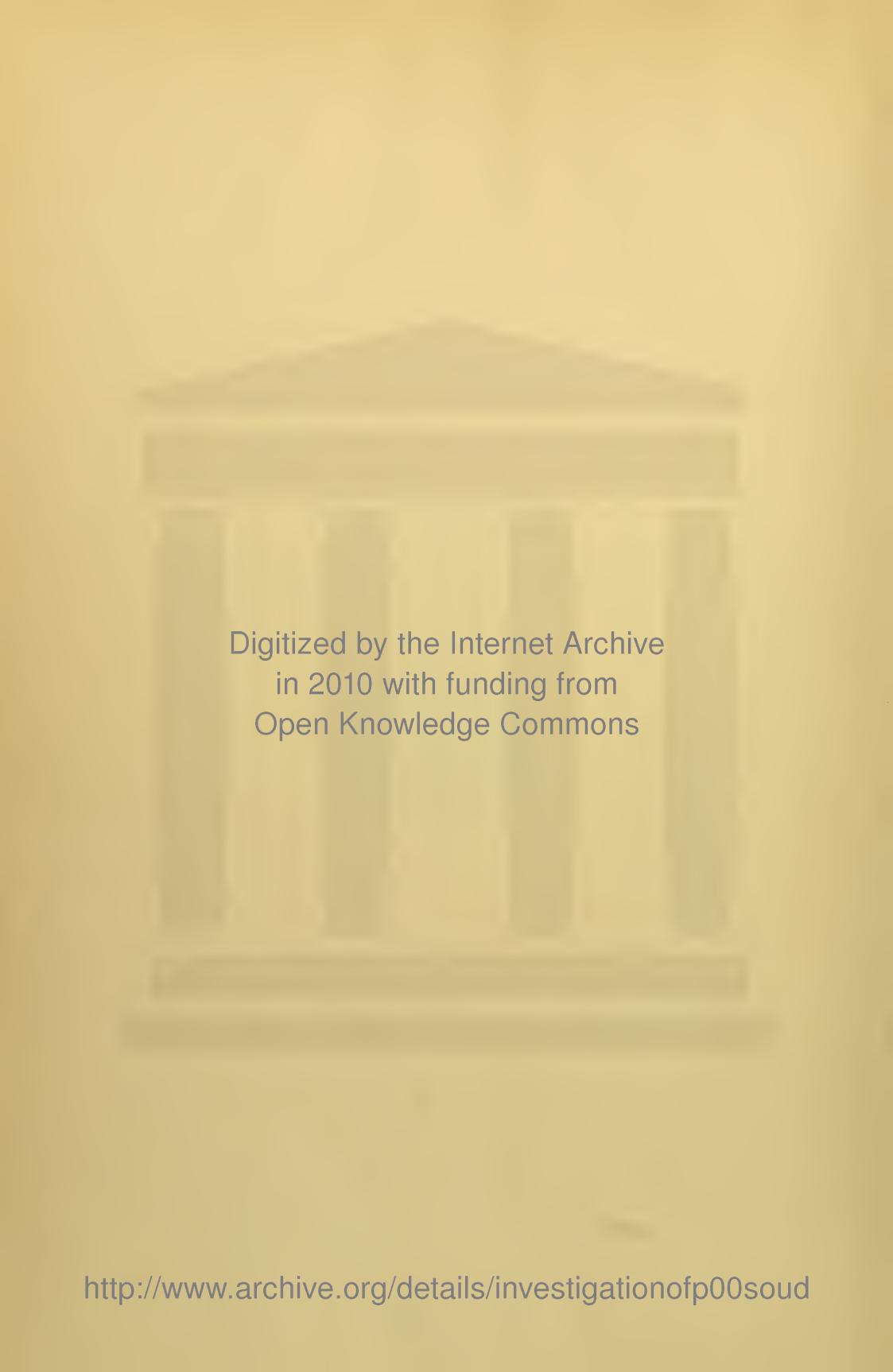
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By Wilmer H. Souder and Chauncey G. Peters

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I. INTRODUCTION

A recent request from a branch of the Government, purchasing large quantities of dental supplies, resulted in the authors taking up a systematic study of the physical properties of certain filling materials together with the instruments generally used in testing the same.

Properties, such as crushing strength, flow, thermal and chemical expansions, chemical composition, electrode-potentials and thermal reactions have been investigated. Additional phases, such as method of manipulation, time, temperature, etc., were found to exert definite influences in certain tests and indicated the necessity for their proper control.

It is believed that the discoveries in instruments will be of sufficient importance to justify lengthy descriptions since it was found necessary to discard practically all devices described in the dental text books. In the selection of instruments we have considered two features, viz, accuracy and simplicity.

Some of the alloys included in this research were made to requested specifications, others were purchased in the open market, and many were submitted by manufacturers cooperating in the investigation.

Many of the results should be interpreted as comparative or relative tests. Since it is not our purpose to advertise the merits or demerits of any material, the manufacturer's names have been omitted. Each has, however, been informed of the results found for his alloy. The purpose of this paper, as in all previous reports, is to place before those familiar with the use of dental materials accurate and reliable data, together with a description of instruments suitable for measuring the properties investigated.

We have attempted to discuss some of our results on a purely physical basis. Their clinical interpretation is left to those experienced in the profession.

It is hoped that at a near date and with the cooperation of the manufacturers and users of these materials it will be possible to write definite and proper specifications, which will enable the purchaser and user to secure articles of known qualities.

1. HISTORICAL

It is practically impossible to give a complete bibliography of the researches on amalgam during its 75 years of existence.

The first amalgams were manipulated in a crude and empirical way, thus entailing an unusually large number of failures. In fact, the feeling against amalgam was so strong at one time that any one speaking favorably of its possibilities was immediately boycotted by the profession. Is it any wonder that failures were very conspicuous when we read of the crude practices and lack of definite data on the manufacture and manipulation of alloys? Opinions and speculations seem to have been considered of more importance than data or accurate statistics.

Despite the failures the few successful restorations were so completely satisfactory that investigators started a search to find the essential properties and technique incident to satisfactory restorations. The works of the pioneers, Tomes, Fletcher, Hitch-

cock, Witzel, and Flagg, have been reviewed and elaborated by Black, who has probably done most to point out the necessity for accurate scientific tests. The chapter on amalgams in Volume II of Black's *Operative Dentistry* (1914) contains a world of information on early work, together with the results of his own extensive researches.

More recent laboratory researches are recorded in the various dental journals under the authorship of Marcus L. Ward, dean, U. of M. Dental School; A. W. Gray, research director, L. D. Caulk Co.; W. G. Crandal, Spencer, Iowa; C. M. McCauley, Abilene, Tex.; William E. Harper, Chicago, Ill.; A. Fenchel, Hamburg, Germany; B. R. Bakker, Utrecht, Holland; and McBain & Joyner, Bristol, England.

The findings of these men are not concordant in all essentials and the lack of definite descriptions of instruments and manipulative details makes it difficult to explain all the results reported. It is hoped that the present article may clear up some of the points in question among the various investigators and also enable those interested in continuing the work to proceed with apparatus of unquestionable accuracy and simplicity.

II. INSTRUMENTS

The first instruments of importance for systematically testing amalgams were those of Dr. Black's design—the micrometer and dynamometer. These were of the utmost value in securing qualitative information and started a new era in amalgam manufacture, but they can scarcely be relied upon to give the most precise or decisive data. Few, if any, investigators are using them to-day. The writers are familiar with the apparatus used by Gray at Milford and by Ward at Michigan and feel sure that both types are an advance over the original apparatus of Black and quite accurate for their researches.

1. THE BLACK MICROMETER AND WEDELSTAEDT TUBES

Fundamental errors precluding the use of the Black micrometer for precision work are the mechanical impossibilities (to date) of combining a system of levers and gears to operate a dial or mirror indicator which will accurately or consistently measure directly to

$\frac{1}{10,000}$ inch ($2\frac{1}{2}$ microns). Scale divisions on an instrument are not always an indication of its approximate sensitivity, although

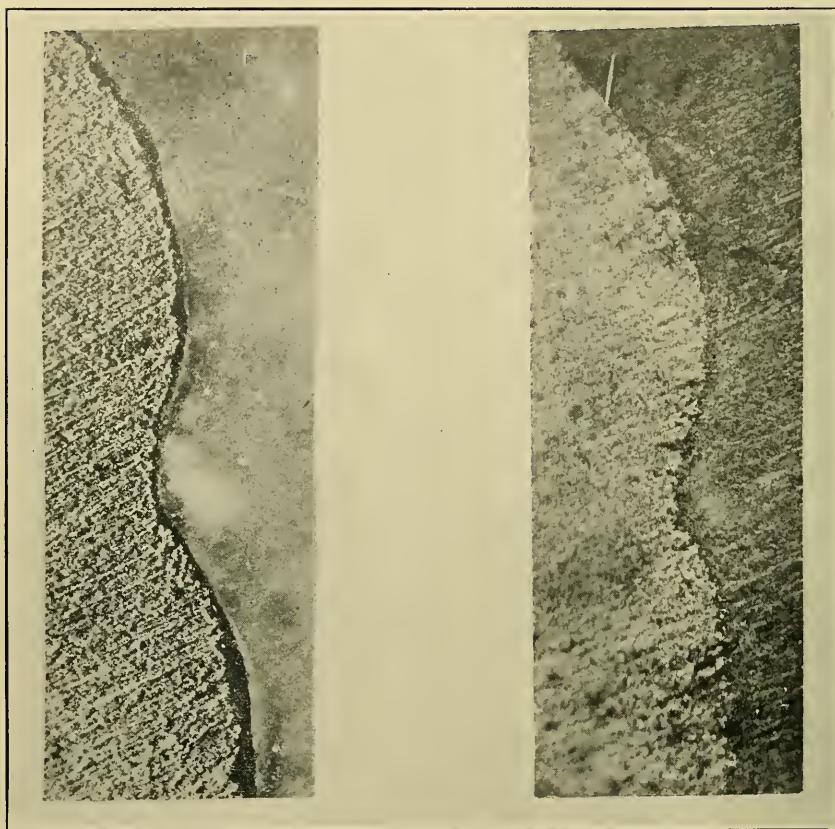
they should be. The elements of variance, passivity, and backlash always enter where there is a multiplicity of moving parts. These features, as affecting instrument design, are fully treated in an article by F. J. Schlink, Scientific Paper No. 328 of this Bureau.

The Wedelstaedt tubes used with this micrometer are subject to criticism because of their restraining action on the amalgam specimen. Later it will be seen that, as shown below, the expansion of amalgam is about two and one-half times as large as steel; hence on the least temperature rise the amalgam is frictionally locked against the sides of the tube and in many cases the excess expansion causes a bulging or "spheroiding" of its free surface. The same phenomenon will occur with chemical or crystallization expansions. Any later temperature drop will be accompanied by the larger contraction of the amalgam (see section on thermal expansion), thus giving the "black-ditch" effect. A slight heating of one of these tubes of amalgam is sufficient to cause an apparent excessive expansion or spheroiding followed by the ditch effect.

Again the "points" expansion are not in the same units as "points" contraction, since in the latter case the amalgam may shrink in all dimensions; whereas in the former the three dimensions of expansion have been forced into a threefold one dimension of expansion, two of which are now manifest as flow, but recorded as expansion. This probably accounts for the claim of slight expansion and no contraction which is often made for alloys; whereas in fact there is contraction which is too small for detection with such apparatus.

2. THE BLACK DYNAMOMETER

Little need be said about the dynamometer except that the results are of necessity irregular, due to the smallness of specimens. This smallness of specimen permits undue influence from slight variations of manipulation, mixing, or packing. Cubical forms are not well suited for these crushing tests. The exposed corners and equal dimensions are factors which should be avoided. For the proper test of compressibility, as outlined by the American Society for Testing Materials, specimens should be prepared in cylindrical form, 1 inch in diameter and $2\frac{1}{2}$ to 4 inches long. Special attention is directed to the necessity of using specimens with a length greater than the diameter in order to avoid the "barrel" effect in case there is a tendency toward flow.



a

Photo by R. E. Lofton, mag. $\times 55$

b

FIG. 1.—*Amalgam-enamel margin*

Same margin viewed by different illuminations. Wrong interpretations often result from an improper use of the microscope

3. THE FLOW ATTACHMENT

Some types of dynamometers are equipped with an auxiliary flow dial which records the motion of the compression rod as the pressure is applied for crushing. The objections offered above apply equally well against this attachment as an instrument of precision.

The sudden shock given the levers and dial parts at the instant of rupture is sufficient to disturb the bearing surfaces and adjustments of all parts of the instrument.

4. OPTICAL MICROMETERS AND COMPARATORS

Unless handled by a person skilled in their use these instruments are of little value. The accompanying figure is included to explain the error possibilities of devices depending upon microscopes; (a) and (b), Fig. 1, are photographs of the same tooth, with absolutely no changes of position of microscope or tooth, the only change being a slight modification of the illumination. In the first instance there is no question about the imperfect adaptation of amalgam to tooth; in the second it appears reasonably perfect. The differences are even more striking when using the binocular microscope.

Micrometer microscopes are often used in comparing and calibrating line (length) standards. These, when arranged for the best possible accuracy—that is, best illumination and most suitable lines on a properly surfaced background—will give results agreeing within 1 micron. However, if only a single microscope is used and the displacement measured in terms of the run of a screw, settings having been made with the assistance of a microscope, then it is necessary to add to the uncertainties of the microscopic setting the errors and irregularities of the screw. A third source of error lies in the necessity of using an auxiliary contact to transmit any motion of the amalgam, since the new amalgam surface is constantly changing its character and necessarily also the character or appearance of any line or mark which may have been placed thereon.

The combined errors make it difficult, if not impossible, to detect variations of 1 or 2 microns, and again the slight contractions of samples in Wedelstaedt tubes may pass unnoticed.

Optical lever devices read by the mirror and scale method are only slightly, if any, better. Their use necessitates a multiplicity of contacts and bearing surfaces, each of which is subject to its

own peculiar action, and each introduces its inherent source of error.

The safer way to proceed in using any of the above instruments is to use samples of amalgam of larger dimensions, thus magnifying the effect and consequently increasing the accuracy of values.

Probably the best instrument for rough measurements is an ordinary small screw machinist micrometer adjusted for a specimen about 50 mm long. This instrument is reliable to better than $\frac{1}{100}$ mm ($\frac{1}{5000}$ inch), about 2 so-called Black points; but the specimen being about six times as long as the Wedelstaedt specimen there results a net increase in theoretical accuracy. Such an outfit properly handled in a thermostated chamber will be of more decisive value than many of the above-described instruments, which are used in looking for ditches or bulges and depend entirely upon the magnification of margins.

III. MANIPULATION OF ALLOY

This subject is often lost sight of in the discussion of physical properties. Probably no other phase of the restoration is subject to greater carelessness. The dentist may spend unusual care in shaping the cavity after having removed a generous amount of good tooth structure to make sure the last traces of decay have been eliminated and then proceed to make up the amalgam on the assumption that the only requirement is to daub up the cavity. Or if the assistant is doing the amalgamation and starts too soon, he is instructed to keep working the mass until the cavity is ready. Another practice is to keep the mass plastic by the addition of slight amounts of mercury at intervals, thus making it possible to condense several cavities from the same mix. The serious effects of this over trituration will be found in another section.

The amalgamation and condensation procedure throughout this research (unless otherwise specified) was according to manufacturers' instructions. In the absence of definite instructions the following technique was adopted and is not seriously different from the majority of manufacturers' instructions.

The alloy and mercury were weighed in an approximate ratio, such that there would be a very slight excess of mercury on condensation. These were mixed (not ground) in a mortar for one minute. The amalgamated mass was then transferred to the hand and mulled two minutes. Part of the excess mercury was removed through chamois cloth. The artificial or matrix cavities were holes in steel blocks drilled and polished. The amalgam was

condensed in these, using three or four different-sized pluggers. Those specimens which failed to show perfect adaptation to the form were rejected. Sufficient alloy was used so that an excess of amalgam could be built up over the top of the cavity, and on tapping lightly it was usually possible to cause additional mercury to rise from the cavity. This excess mercury and amalgam was later removed and the specimen taken from the matrix. The condensation was usually completed in two or three minutes, depending upon the size and shape of specimen.

The slight excess of mercury used appeared to give the condensed amalgam a greater uniformity and prevent the formation of layers, when added quantities of amalgam were condensed in the cavity. These layers are fostered by delay in condensing, hence the desirability of using shorter times.

The greatest variation in results, when using a given manipulation, appears to arise not so much from slight irregularities in manipulation of alloy as from variation in alloy from package to package. All values reported for alloy *G*¹ are for the same package; this is also true for alloy I. A fair degree of agreement will be found for check tests on these alloys recorded in Table 3. Another method of securing uniformity of results resorted to by some investigators consists in packing specimens under continued mechanical pressure, allowing several minutes to elapse before removing them from the matrix. The authors have been more interested in finding what happens to amalgams as they are ordinarily used, or should be used, and have made an attempt to start tests early enough to discover all changes accompanying the reaction, regardless of variations.

IV. DIMENSIONAL CHANGES WITH TEMPERATURE

1. APPARATUS AND METHODS

For the measurement of the thermal expansion of small samples, methods and apparatus which make use of the interference of light waves have been employed at this Bureau for several years. For some of this work the old-established Fizeau² method was used. Fig. 3 shows an interferometer devised by Priest³ for determining the thermal expansion of single small pins. With

¹ Letters have been assigned to the trade names of the alloys and throughout this paper the same letter refers to the same alloy, but not necessarily to the same package of alloy except in the cases mentioned above.

² Fizeau, Annal de Chem. et al Phys. (4) 2, 143; 1863.

³ Priest, B. S. Scientific Paper No. 365, 1920.

another form of apparatus, recently described by one of the authors,⁴ the expansion of three different pins can be simultaneously determined. These interferometers have been thoroughly tried out and found to measure small displacements with an error of about 0.005 micron or 0.0000002 inch. Because of the extreme accuracy and directness of the method and the necessity of using small samples, the interferometer was used for the determinations of thermal expansion of teeth and dental materials. A rather complete description of the apparatus has been given here for those who may be interested in making measurements of this kind.

The interferometer is usually thought of as an extremely complicated apparatus, while in reality the essential parts are two

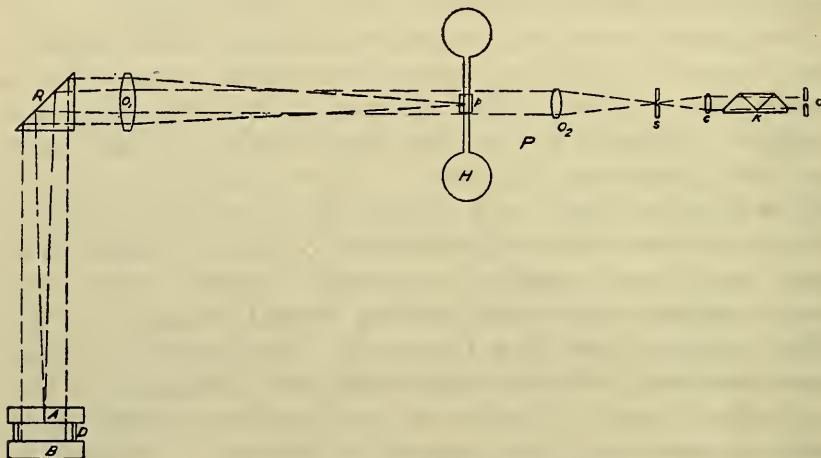


FIG. 2.—Vertical section of apparatus

plane glass plates which are held apart by a suitable separator. Fig. 2 shows a vertical section of the apparatus.

The two plates *A* and *B* and the ring *D* constitute the interferometer, while *P* represents a Pulfrich apparatus for viewing the fringes.

The light from a helium lamp *H* is focused upon a small total reflection prism *p*. After being collimated by the lens *O*₁, it is reflected by the prism *R* down to the interferometer plates *A* and *B* which are in the focal plane of *O*₁. The rays returning from points in the plane of the mirrors are collimated by the lens *O*₁, and an image of the interference pattern and the reference marks on the plates is formed by the lens *O*₂ upon the slit *S* and viewed

⁴ Peters, Jour. Wash. Acad. of Sci., 9, p. 281; 1919.

with the eye piece *C*. The direct vision prism *K* separates the fringe patterns due to the helium light of different wave lengths.

A more detailed view of the interferometer is shown in Fig. 3. The upper surface of the base plate *B* is a polished true plane. One side of the plate which projects about 0.3 mm above the upper surface of *B* forms a knife edge *SS* parallel to that surface, while the other side is undercut, leaving the edge *EF* parallel to *SS*, and the base *M* which forms a support for the sample *x*.

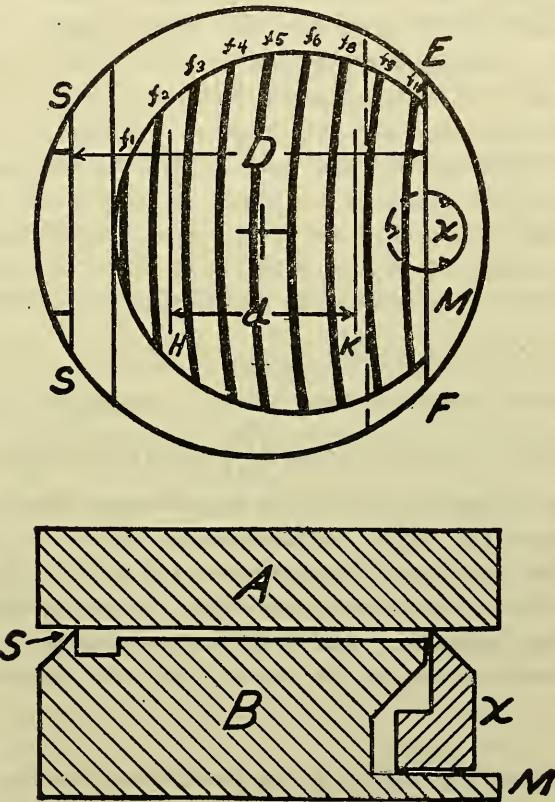


FIG. 3.—Interferometer

The upper end of the sample is cut away to the center from one side and bevelled to an edge from the other. The flat surface of the sample is placed in contact with the edge *EF*, thus assuring a constant distance *D* between the sample and the knife edge *SS*. Two reference lines *H* and *K*, distance *d* apart, are ruled parallel to the knife edge *SS* on the upper surface of the base plate.

The upper interferometer mirror *A*, which is a plate of glass with both faces polished true plane, rests on the knife edge *SS*

and the top of the sample x , adjusted to be slightly higher than SS . A narrow wedge-shaped space is thus formed between the two plates the widest part of which is toward x . Considering the two faces of this wedge, light from the lamp H made parallel by the lens O_1 , Fig. 2, falling upon the lower surface of A , Fig. 3, is in part reflected and the rest transmitted to the upper surface of B . Here again part of the light is reflected. Between these two reflected wave trains "interference" takes place. The observer, viewing this reflected light, sees straight dark bands parallel to each other across the face of the wedge. In Fig. 3, f_1 , f_2 , f_3 , etc., represent these bands. The band f_1 shows that along that line the distance down and back between the plates is some whole number of wave lengths. On moving to a wider part of the wedge another line f_2 is reached where twice the distance between the plates is one wave length greater than at f_1 . Similarly along f_3 this distance is two wave lengths greater than at f_1 , etc. Starting from f_1 each successive band denotes that the separation of the two plates has increased by one-half the wave length of the light or about $\frac{1}{100\,000}$ of an inch. Therefore the total number of bands between SS and x multiplied by one-half the wave length of the light used $\left(\frac{\lambda}{2}\right)$ gives the difference in separation of the plates at x and SS or the difference in elevation of x and SS above the upper surface of B . As it is difficult to determine the number of bands between x and SS , the number N between the two reference lines H and K is determined. The corresponding distances D and d are known, therefore the difference in elevation of x and SS above B at any time is equal to $\frac{\lambda D N}{2d}$.

Let L_x denote the length of the sample and L_s the length of standard material between SS and the plane of M . Let ΔL_x and ΔL_s represent the elongation of L_x and L_s caused by a rise of temperature ΔT . If L_x and L_s expand differently the relative elevations of x and SS change, causing a change in the number of bands between H and K . Let N_1 represent the number of bands between the reference marks before and N_2 the number after the temperature change takes place; then,

$$\Delta L_x = \Delta L_s + \frac{\lambda D}{2d} (N_2 - N_1) \quad (1)$$

That is, the elongation of x is equal to the elongation of s plus the difference in elongation of the two, which is determined from the change in the number of bands between the reference marks.

The coefficient of linear thermal expansion is the elongation of unit length per degree rise of temperature

$$C = \frac{\Delta L}{L \Delta T} \quad (2)$$

The coefficient of expansion of x is then given by the expression

$$C_x = \frac{\Delta L_x}{L_x \Delta T} = \frac{\Delta L_s}{L_x \Delta T} + \frac{\lambda D (N_2 - N_1)}{2d L_x \Delta T} \quad (3)$$

Since L_x and L_s are practically equal this becomes

$$C_x = C_s + \frac{\lambda D (N_2 - N_1)}{2d L_x \Delta T} \quad (4)$$

Where C_s is the coefficient of expansion of the base plate material.

In order to control the temperature properly the interferometer was mounted on a steel block G in the bottom of the container C , Fig. 4, which consisted of a steel tube, 30 cm long, 5 cm in diameter, and 1 mm thickness of wall, the upper end of which was closed with a glass window W_1 ; 10 cm from the base another glass window W_2 was supported by a heavy brass ring. Most of the lower part of the tube was cut away to allow easy adjustment of the interferometer. This end of the tube was closed with a steel cup R which screwed onto the tube at U with a rubber gasket to make the joint tight. A little mercury in the bottom of the cup made good metallic contact between the base block and the bath. The container was lowered with a rack and pinion into the oil bath which regulated the temperature. The bath liquid was circulated through the tubes D and E by the propeller P . To eliminate vibration the motor M was supported on a separate base and connected to the propeller shaft by a small rubber tube. The liquid was cooled by the brine coil K and heated with a 10-ohm resistance coil H the current in which was regulated by a relay operated by the thermostat J . This apparatus was thermally insulated with ground cork and mounted in a wooden box.

The temperature of the liquid around C could be held for any length of time within 0.01° of the desired value. The tempera-

ture of the bath was read with a thermometer T and a five-junction thermocouple L , and the temperature of the base block and sample with another five-junction thermocouple N . A temperature survey was made of this apparatus with differential

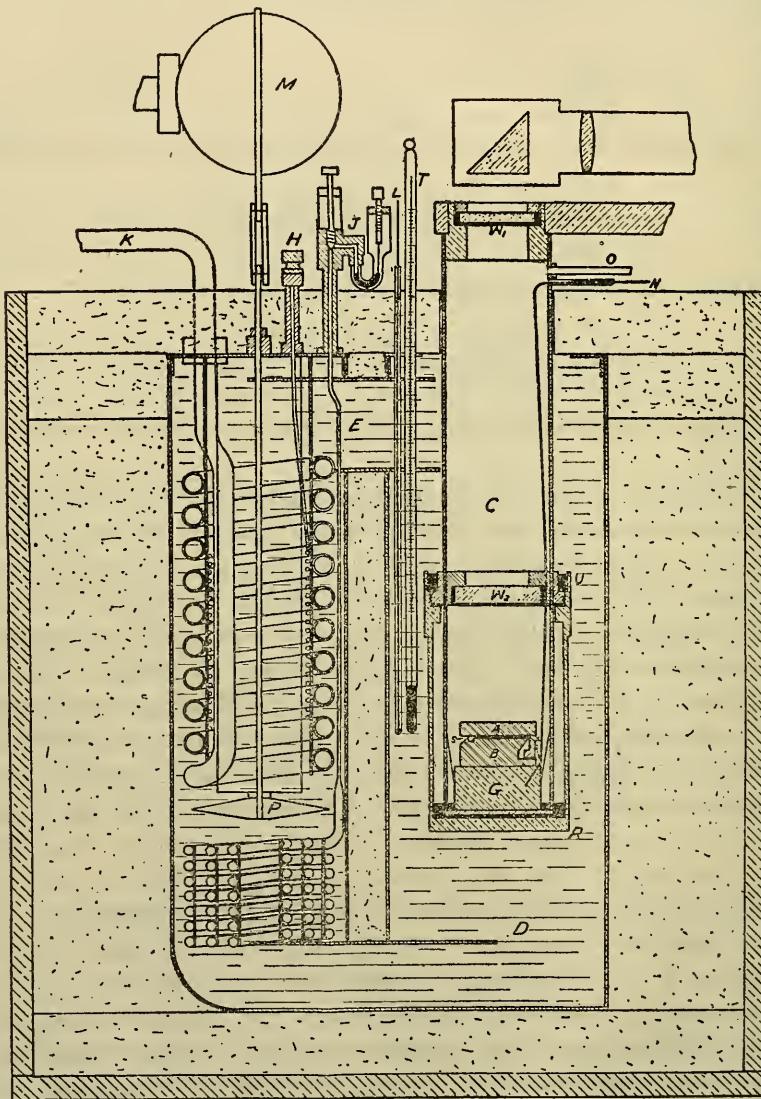


FIG. 4.—Temperature control apparatus

thermocouples to determine both the time required for the interferometer and sample to reach a steady state when the temperature of the bath was held constant, and the lag of the sample when the bath temperature was changing.

2. EXPERIMENTAL PROCEDURE

The amalgam material after being prepared in the manner already described was made into samples about 6 mm in diameter and 1 cm long, illustrated by x , Fig. 3. These samples were kept for several weeks before measurements were made. The porcelain samples were made in the same form and kept under water. The tooth specimens were taken from different teeth and different parts of the same tooth. One of the samples, the expansion of which is shown in Fig. 5, was taken from the crown of a large molar, the other from the root of a long cuspid.

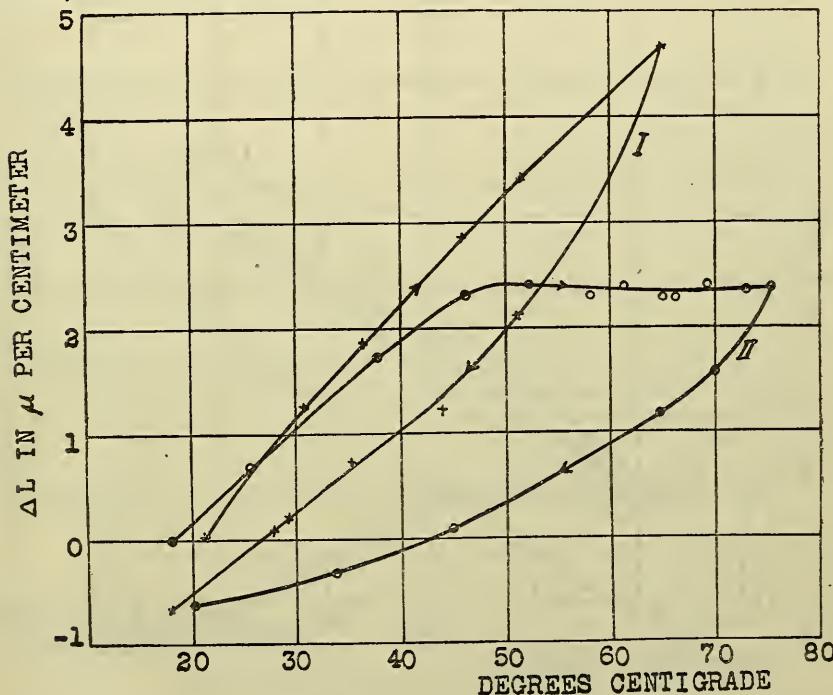


FIG. 5.—Thermal expansion of teeth

(I=crown. II=root)

After the sample was properly adjusted the interferometer was placed in the container and lowered into the bath. Two procedures were followed in making the measurements. The first was to hold the temperature of sample constant for at least one hour at each point before the measurements were made. The second was to change the temperature of the sample slowly, about 1° C in three or four minutes, and make observations periodically.

With the samples from teeth it was found that as soon as they were heated moisture evaporated and a very rapid contraction

took place amounting to 0.2 to 0.3 mm for a 1-cm sample. These samples regained their original length if allowed to stand in water for two or three days. To overcome this contraction, which was many times greater than the thermal expansion of the material, the interferometer and sample were placed in a container filled with water. This container was then placed in the temperature-control apparatus. With both the porcelain and teeth it was found necessary to keep the samples under water and make the measurement with the samples under water in order to obtain

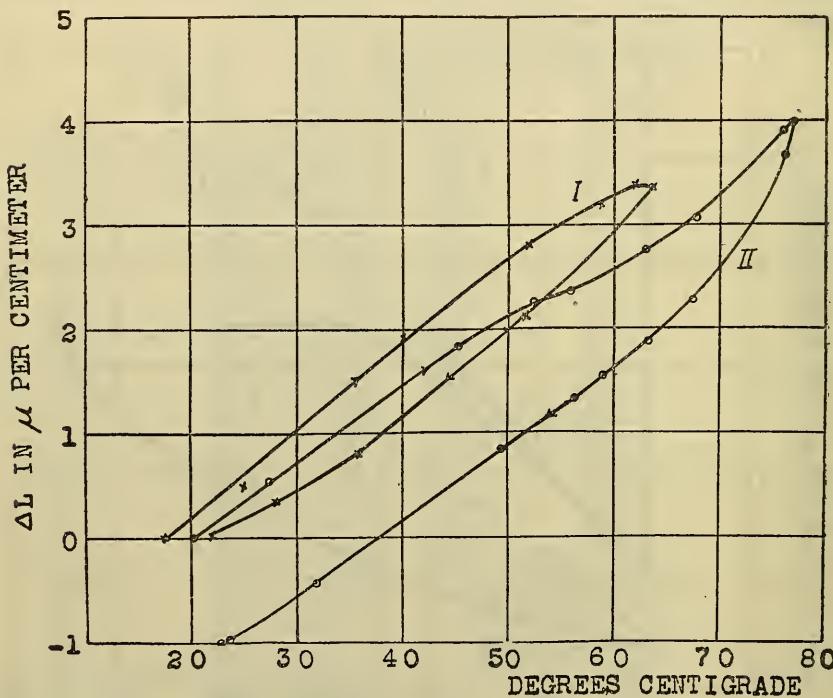


FIG. 6.—*Thermal expansion of synthetic porcelain*

consistent results. This procedure seems to reproduce actual condition of the mouth and yield values of thermal expansion as near correct as it is possible to obtain with this type of material.

3. RESULTS

The following curves represent the elongation of the different materials with change of temperature. Degrees centigrade are plotted as abscissae and change in length (ΔL) in microns (μ) of a sample 1 cm long as ordinates. All the observations that were taken have been plotted, none were rejected or omitted.

Curve *I*, Fig. 5, is for a sample cut from the crown of a molar, Curve *II* for a sample from the root of a newly extracted cupid.

Curves *I* and *II*, Fig. 6, represent the expansions of two different samples of porcelain.

The curves in Figs. 7 and 8 are for different kinds of amalgams. These curves are lettered to correspond with kind of material from which the samples were made. In fact, most of the samples had previously been used for experiments of setting changes.

Most of the samples showed irregular behavior near 80° C. Some of the samples when removed from the container were

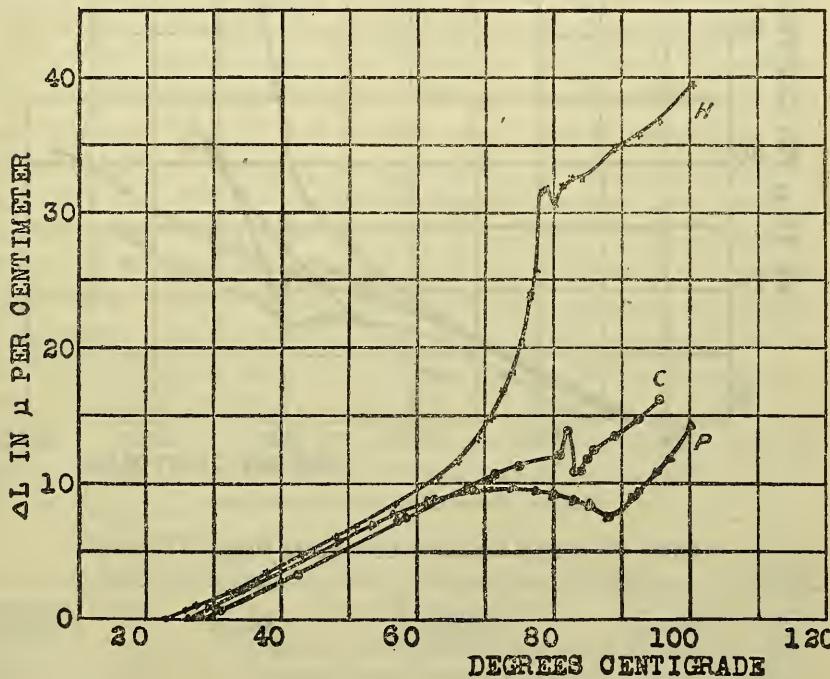


FIG. 7.—Thermal expansion of amalgam

covered with soft drops of mercury which formed into bright crystals in a short time. An inspection of these curves and the samples showed that the amalgams must have undergone some radical transformations when heated. It is very probable that any filling or part of a filling, if by chance subjected to temperatures near this value, may suffer serious injury or have its physical properties entirely changed.

In Table 1 there are tabulated data on the linear thermal expansion of a number of materials, including sections of teeth.

From this table it is seen that the expansions of teeth range from 6.4 to 11.4, depending upon the tooth selected and the portion of the tooth used, an average value of approximately 8. The porcelain average value is not far from the above figure, gold is somewhat higher (14.4), while the amalgams average about 25.

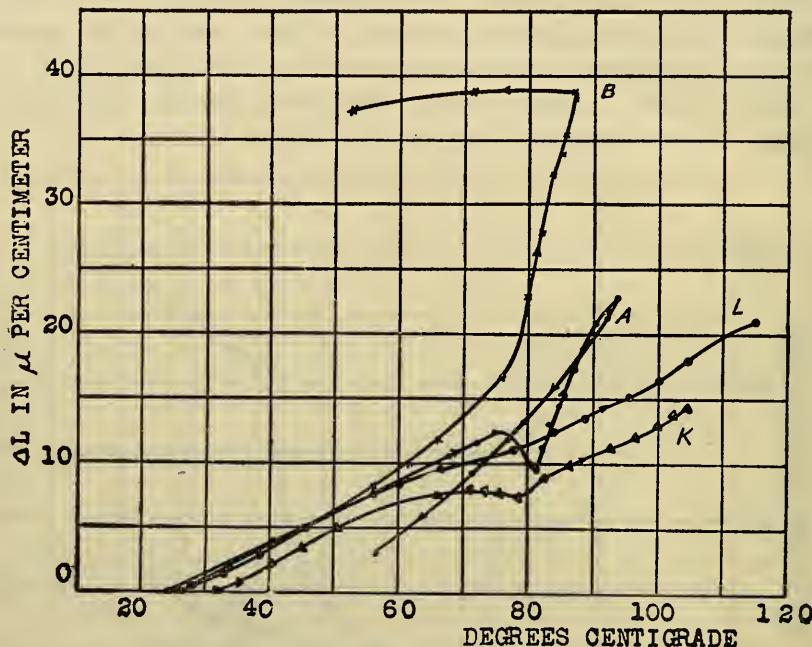


FIG. 8.—*Thermal expansion of amalgams*

TABLE 1.—Average Expansion Coefficients, Range 20 to 50° C

Material	Expansion coefficients x 10 ⁶	Material	Expansion coefficients x 10 ⁶
Tooth (root).....	8.3	Amalgam (C).....	25.0
Tooth (across crown).....	11.4do.....	24.7
Tooth (root and crown).....	6.4do.....	28.0
Do.....	8.7	Porcelain (Bayeux).....	4.1
Do.....	8.3	Gold.....	14.4
Synthetic porcelain.....	7.1	Platinum.....	9.0
Do.....	8.1	Silver.....	19.2
Do.....	7.5	Mercury (linear).....	60.6
Amalgam (H).....	26.4	Zinc.....	29.2
Amalgam (C).....	25.0	Tin.....	22.3
Amalgam (K).....	22.1	Copper.....	16.8
Amalgam (P).....	24.5	Gutta-percha.....	198.3
Amalgam (A).....	25.4	Aluminum.....	23.1
Amalgam (B).....	28.0	Steel.....	11.0
Amalgam (L).....	24.8		

Since the effects due to the differential expansions of the materials depend upon the temperature range, size of cavity, and elasticity of tooth substance and material, no general statement should be made regarding the relative merits of materials unless all the important conditions are given due consideration.

To illustrate, let us consider the case of a maximum cavity and filling 1 cm in diameter, undergoing a temperature variation of 50° C. Then the free expansion along each coordinate axis is 4 microns for the cavity, 7 microns for the gold filling, and 12.5 microns for the amalgam. (If the dimension or temperature range is less, these effects will be proportionally reduced.) If we have perfect adaptation and no stress at the lower temperature, then at the higher temperature there are two extreme possibilities: (a) The elasticity of the tooth and compressibility of the material may be such that perfect adaptation is maintained, or (b) the rigidity of the tooth and plasticity of the material may be such that there will be a flow of material in the only free direction causing a spheroiding or bulging over the cavity. With a perfectly rigid tooth cavity this may equal three times the linear differential expansion of the substances, which will be 25 microns in the case of amalgam. Should the filling material take a permanent set at the higher temperature, then on returning to the lower temperature, all materials having undergone free contraction, there is a possibility of a 4-micron separation around the filling. This is analogous to the case of heating the Wedelstaedt tube referred to in a previous section.

The above temperature range of 50° C has been decided upon as a fair representation of the temperature variation to which metallic fillings may be subjected. In order to test this experimentally, copper-constantan thermocouples were cemented into amalgam inserts 1 to 3 mm from the surface, and the temperatures of the inserts were read by means of a potentiometer when different foods and drinks were taken into the mouth. With ice water and cracked ice temperatures as low as 5° C and with hot foods and drinks temperatures from 50 to 60° C were observed in the inserts. No unusual or excessive sensation of pain was indicated by the adjacent vital teeth during the experiments.

V. DIMENSIONAL CHANGES WITH TIME**1. APPARATUS**

The apparatus described in the previous section was used to measure the dimensional changes of newly made amalgam samples during the hardening or setting period. The temperature of the sample and interferometer was held constant and the variations in the length of the specimens with time were recorded. These dimensional variations during hardening will be referred to as setting changes.

In equation (1), ΔL_s represents the change in the length of the material under the knife-edge, due to the change in temperature. In the present case the temperature was constant, therefore ΔL_s equals zero and the expression for the setting change of the sample becomes

$$\Delta L_x = \frac{\lambda}{2} \frac{D}{d} (N_2 - N_1) \quad (5)$$

Here the interferometer becomes a lever of the second class with the knife-edge forming the fulcrum, the top of the sample the point of application of the force, and the upper plate, which weighs about 10 grams, the load. This type of lever is far less susceptible to friction and contact error than those used in the moving dial or mirror instruments. The only restraining force on the sample is the constant 5 grams weight of the cover plate.

The results obtained from several samples made from the same material and held constant at temperatures ranging from 8 to 37° C showed that careful temperature control was not of sufficient importance to warrant the use of the constant temperature apparatus.

Four interferometers of different construction were mounted in a semicircle on a table so that the Pulfrich apparatus could be turned from one to another for making observations on the interference fringes. A thermometer was placed close to the interferometers and the temperature of the room was recorded after each observation on the fringes. This arrangement greatly facilitated the work, for four samples were investigated simultaneously, whereas the constant temperature apparatus accommodated only one at a time.

2. EXPERIMENTAL PROCEDURE

The alloys were amalgamated and condensed into the mold according to the procedure previously described. The specimens were immediately removed from the mold and adjusted to fit the interferometer, which required from 4 to 8 minutes. The cover plate was pressed down upon the sample with a force of several pounds to insure good contact at the bearing points and to ascertain whether or not the sample had hardened enough to support the weight of the plate. Most of the samples hardened in a few minutes, but a few like *M* and *N*, Fig. 14, were still quite soft after 60 minutes. The time record started the moment the sample was removed from the mold and observations of the length changes, which commenced immediately after the adjustment of the samples, were taken at given intervals over a period of one to several days. With rapidly changing materials of this kind the number of interference bands between the reference lines was estimated to the nearest 0.1 of a band. Since 0.2 of a band is equivalent to about 0.05μ this estimation gave all the accuracy necessary.

At least three samples from each material were investigated according to the above procedure.

3. RESULTS

The following curves represent length changes which took place during setting. Time in minutes is plotted as abscissae and change of length (ΔL) in microns (μ) of a sample 1 cm long as ordinates. An ascending curve represents an elongation and a descending curve a contraction. The time required to adjust the sample to the interferometer is plotted along the zero ordinate. The mean temperature and maximum variation in temperature of each sample have been recorded with the curve.

In Fig. 9 are represented the setting changes of four samples from material *C*. The samples were all prepared according to our regular procedure but were held at different temperatures after being placed in the interferometer. The temperatures at which the different samples were held during the runs are as follows: Sample *C-1* at 37° C , *C-2* at 27.8° C , *C-3* at 20.4° C , and *C-4* at 8° C .

Fig. 10 represents the data taken on four samples of material *B*. The samples were prepared according to our regular manipulation and held very near to the same temperature throughout the ex-

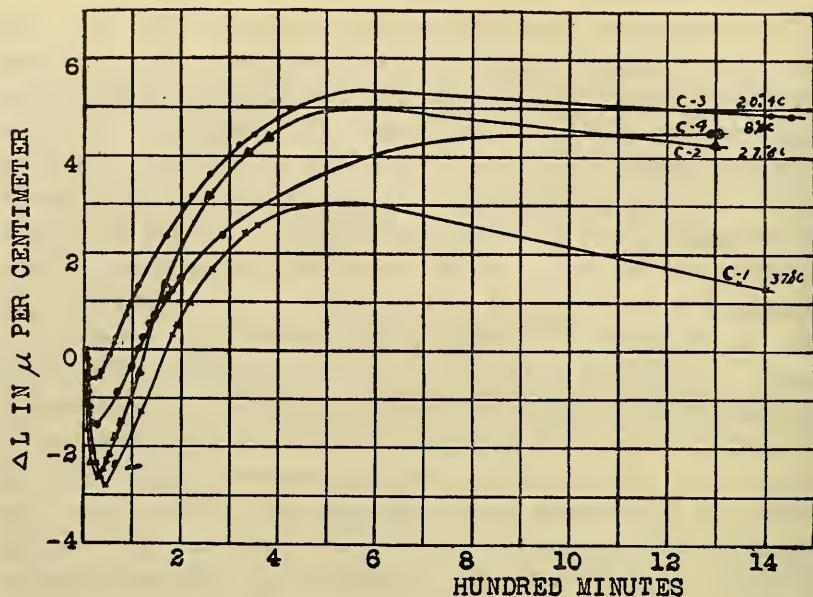


FIG. 9.—Setting changes of amalgam C

Specimens held at different temperatures

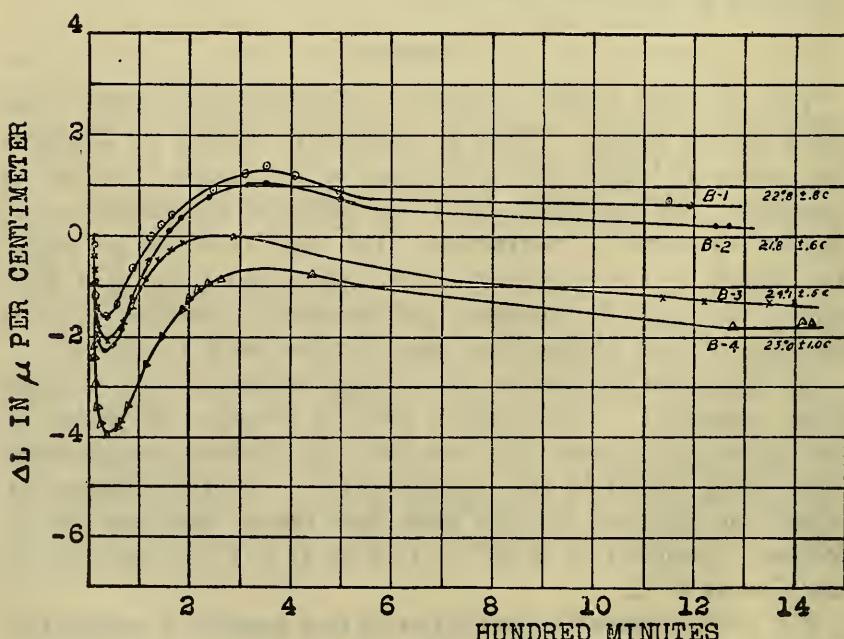


FIG. 10.—Setting changes of amalgam B

Specimens held at nearly the same temperature

periments. The mean temperature of the sample and the maximum variation during the run are recorded with each curve. Figs. 11 to 13 represent the results obtained from samples of alloys *C*, *H*, and *A*, which had been given various treatments before and subjected to similar conditions after condensation. The mulling time was varied from 1 to 35 minutes. A specimen of each alloy was cooked for three hours at 120° C, and 1½ per cent zinc was added to one alloy.

From the foregoing curves it is seen that the length of time devoted to mulling the amalgam exerted by far the greatest effect upon its subsequent behavior. The curves from samples of mate-

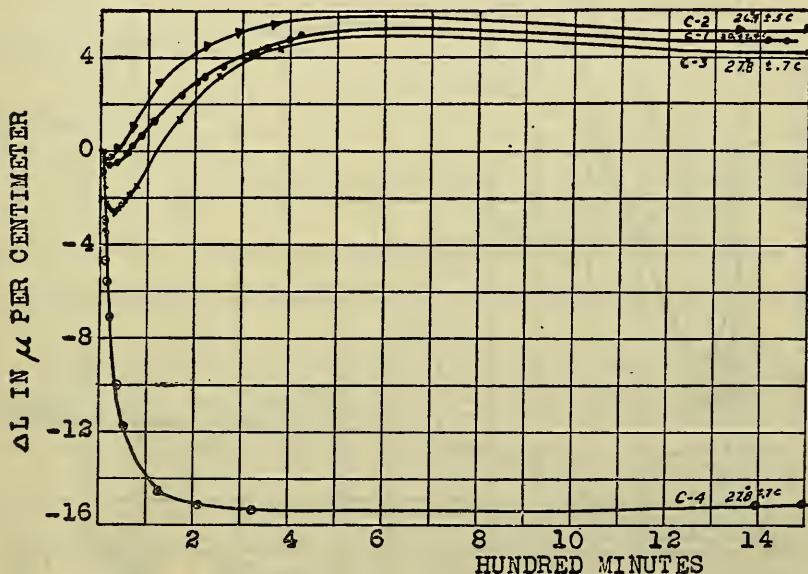


FIG. 11.—Setting changes of amalgam *C*

C-1, Usual manipulation; C-2, alloy cooked 3 hours at 120° C; C-3, mulled 1 minute; C-4, mulled 35 minutes

rial *B*, Fig. 10, which received practically the same treatment, while quite concordant, do not show a much better agreement than those of material *C*, Fig. 9, which were held at different temperatures during the setting period. In Figs. 11, 12, and 13 curve 1 represents the usual manipulation. Curve 2, in which alloys were cooked for three hours at 120° C, differs very little from curve 1. *C-3* and *H-3*, which were mulled only one minute, agree almost as well with curve 1. The addition of 1½ per cent zinc to alloy *A* seems to have changed the character of the setting curve *A-3*. The marked effect caused by mulling the amalgams 20 to 35

minutes is shown by curve 4. Experiments of this kind showed that large setting contractions could be produced in any of the amalgams by overmulling and emphasized the importance of that part of the process. Compared with this effect the slight irregularities due to variations in room temperature seem to be insignificant. If setting contractions of 10 to 20 μ per centimeter are important, then this question of mulling time must be given careful consideration.

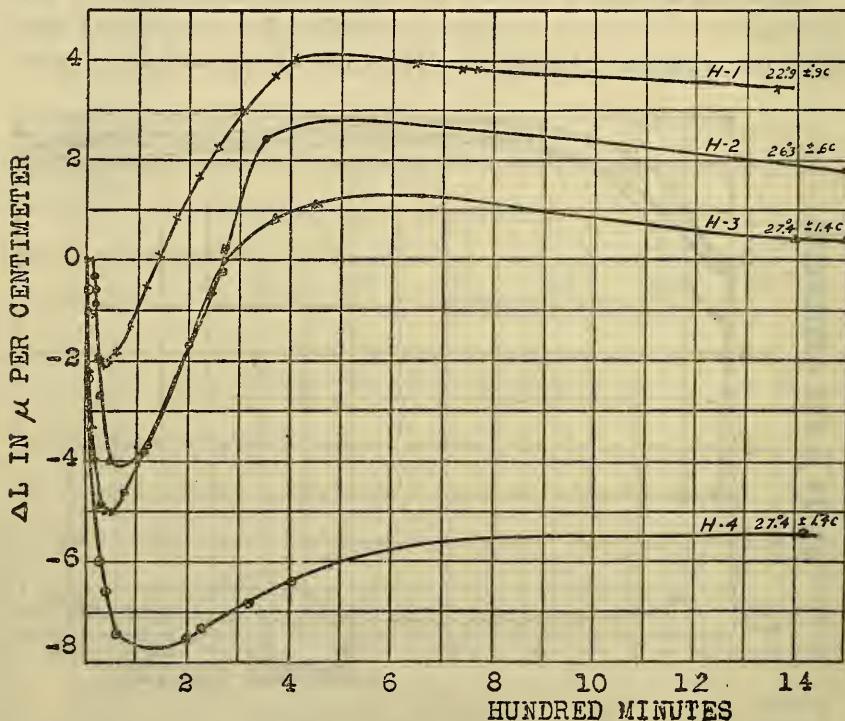


FIG. 12.—Setting changes of amalgam H

H-1, Usual manipulation; H-2, alloy cooked 3 hours at 120° C; H-3, milled 1 minute; H-4, milled 25 minutes

Our results upon effects of mulling agree very well with the findings of Dr. Gray.⁵ He has placed greater emphasis upon the importance of accurate temperature control of the samples during the setting period than we have. Regarding the effect of annealing no comparison can be made because he has neglected to state the temperature at which the annealing was carried out.

The setting changes of each of the other materials are shown in Figs. 14 to 17. At least three samples from each material pre-

⁵ Gray, Journal of the National Dental Association, 6, 10, p. 917; 1919.

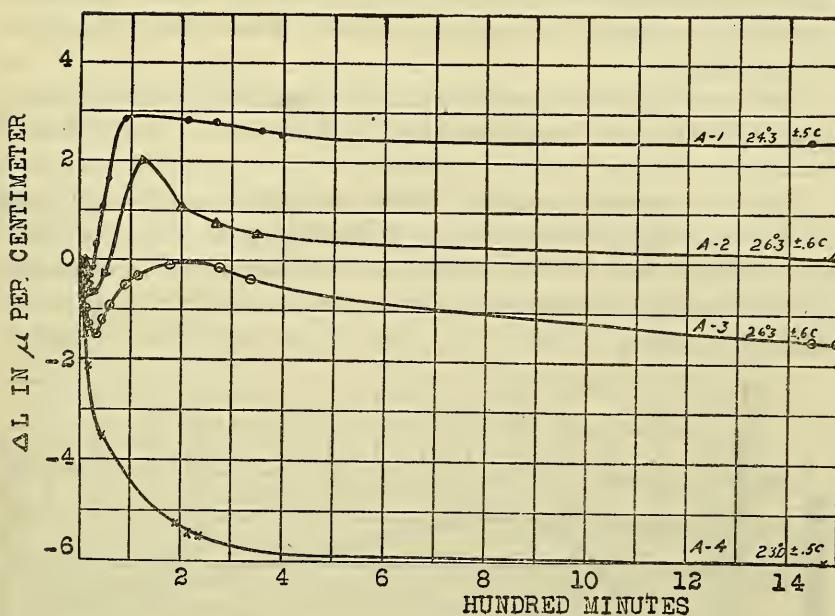


FIG. 13.—Setting changes of amalgam A

A-1, Usual manipulation; A-2, alloy cooked 3 hours at 120°C ; A-3, $1\frac{1}{3}$ Zn added to alloy; A-4, mulled 20 minutes

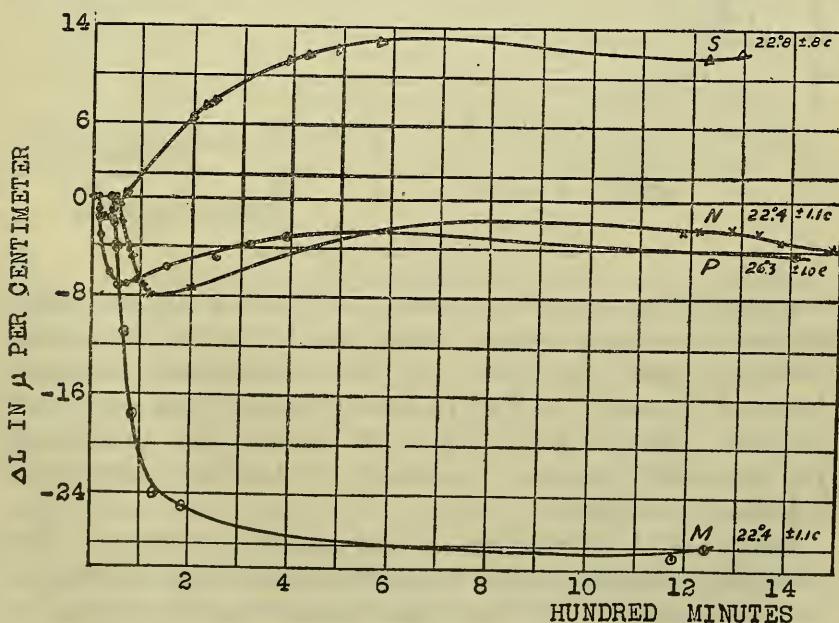


FIG. 14.—Setting changes of amalgams

pared according to the regular procedure were tested and gave concordant characteristic curves similar to those shown for material *C*, Fig. 9.

Only one curve from each material has been plotted for the purpose of making a comparison of the behaviors of the different material.

From the chemical analysis, Table 3, it will be seen that *M* and *N*, Fig. 14, are low silver alloys. These specimens were very slow in setting; in fact, it was impossible to make any measurements during the first hour because the material would not support the lightest pressure. Alloy *S* contained 73 per cent silver. Alloy *P*

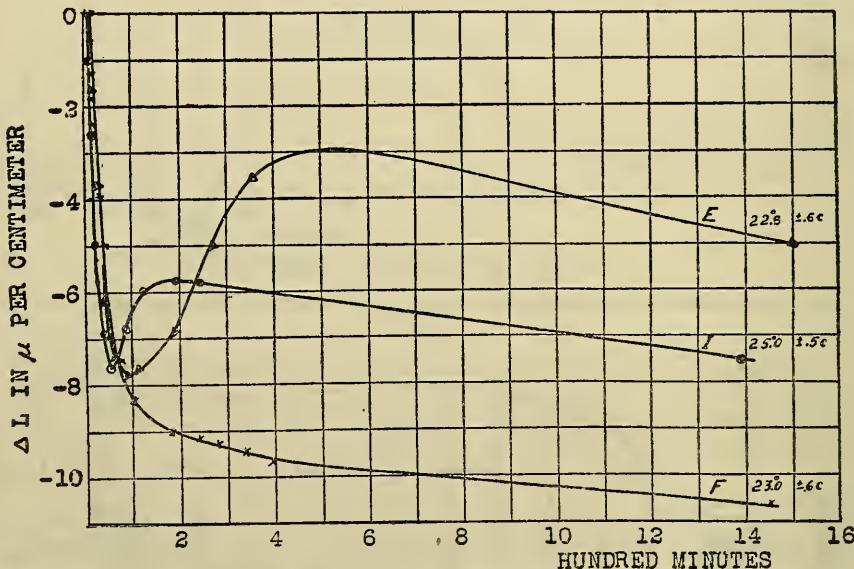


FIG. 15.—Setting changes of amalgams

contained 5 per cent zinc. Alloys *E*, *I*, and *F* are not widely different in setting changes during the first hour, but diverge somewhat after this time. All show permanent shrinkage. Alloys *B*, *G*, and *T* of Fig. 16 have practically the same silver analyses. Alloys *K* and *L*, Fig. 17, are lower in silver than *E* or *F*. The difference in behavior is probably due to relative percentages of two other elements.

Nearly all of the samples show an initial contraction. With some this continued and no recovery was evidenced; with others an initial contraction of from 1 to 3 microns took place in the first 30 or 40 minutes, after which the sample expanded for the next 400 minutes, remaining quite constant during the remainder of

the experiment. The initial contraction could be due to one or all of at least three causes. The sample is usually quite soft for a few minutes, which might cause a settling of the sample under even its own weight; there might be a real contraction of the material; or in adjusting the sample it is heated somewhat in the hand, which is slightly above the room temperature. In the last case a thermal contraction should take place during the first few minutes.

To follow the actual temperatures of the sample during the setting or hardening period, one junction of a differential thermocouple was placed in a block which had the same temperature as the

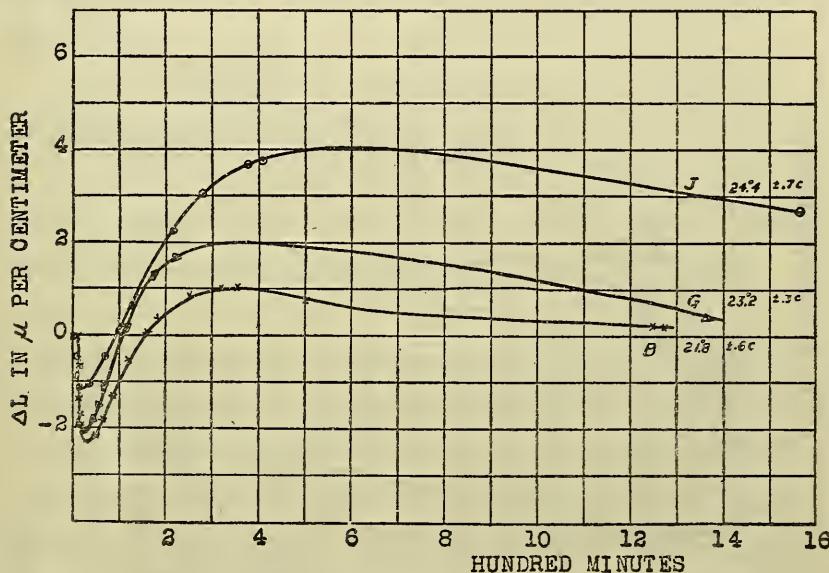


FIG. 16.—Setting changes of amalgams

room. The other junction was packed in the center of the sample, which was made and adjusted according to the usual procedure.

Measurements made immediately after samples were placed in the interferometer showed that their temperatures were 4 to 8° C above the temperature of the block which was very nearly 22.5° C during these experiments. Fig. 18 shows the cooling curves of some of these samples. The sample 19-A was 3.5° C above the temperature of the block when placed in the apparatus and was still 0.7° C above the temperature of the block 150 minutes later. Three hours later it was warmed 8° C above block temperature and allowed to cool. Curve 19-B shows that it returned to the block temperature after 22 minutes.

The second sample was 7.4° C above block temperature immediately after being put into the apparatus. Its cooling is shown by curve 16-A. It was still 0.5° C above block temperature 60 minutes later. The following day its temperature was increased 5° C. It then cooled in 12 minutes as shown by curve 16-B.

Curve 4 shows another sample that required 80 minutes to return from 5° C above to block temperature.

These experiments show that heat is evolved by the material during the amalgamation. This evolution of heat seems to continue during the period while the sample is undergoing most of its variation of length. Of course some of the original heating

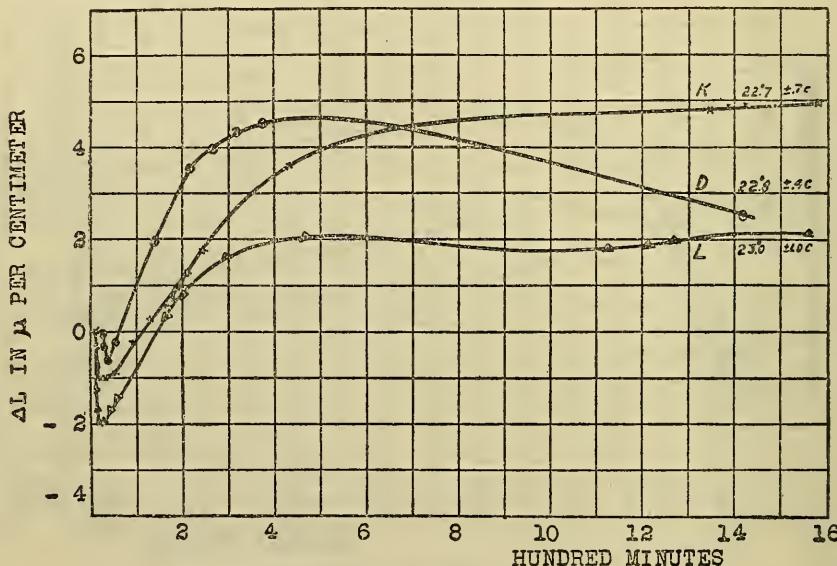


FIG. 17.—Setting changes of amalgams

comes from the hand of the operator, but curves 19-B and 16-B show that the sample should return to the temperature of the room in 15 to 20 minutes. Enough heat seems to be evolved to keep the temperature of the sample above that of the surroundings for several hours. From this it seems that careful control of the sample container does not insure that temperature of the sample is the same as that of its surroundings. Consideration of the foregoing results make it possible to account for 1 to 2 microns of this initial setting contraction by the thermal contraction of the material. The fact that some of the samples, which hardened in a few minutes after condensing, contracted more than that

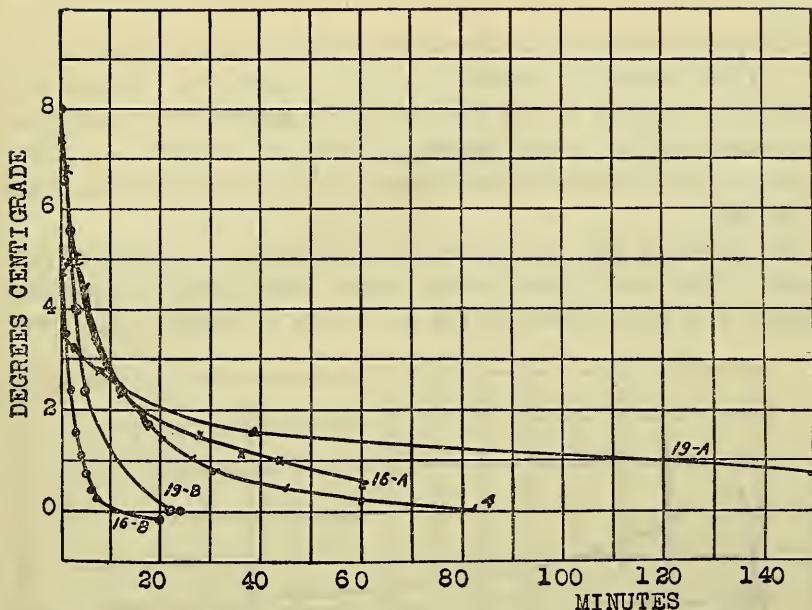


FIG. 18.—Cooling of amalgams during and after setting

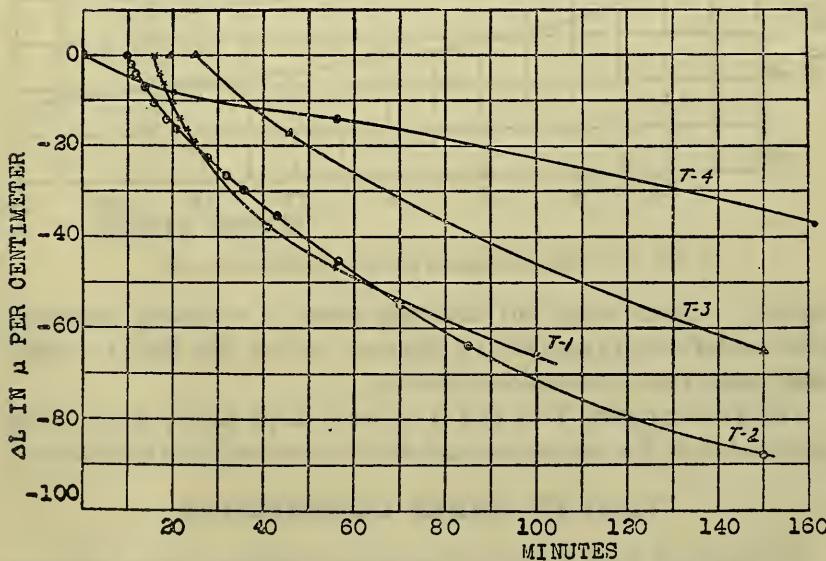


FIG. 19.—Setting changes of synthetic porcelain in air

amount leads us to believe that part of the initial contraction was real.

The porcelain samples contracted rapidly when exposed to the air. This is shown by curve $T-1$ and $T-2$, Fig. 19. Sample $T-3$ retained its length for 25 minutes while under water and then contracted rapidly when exposed to the air. Curve $T-4$ represents the behavior of a sample coated with its varnish and exposed to the air.

The curves in Fig. 20 represent the behavior of samples of porcelain which were kept under water during the experiment. Sample $T-5$ was exposed to the air during adjustment and dried

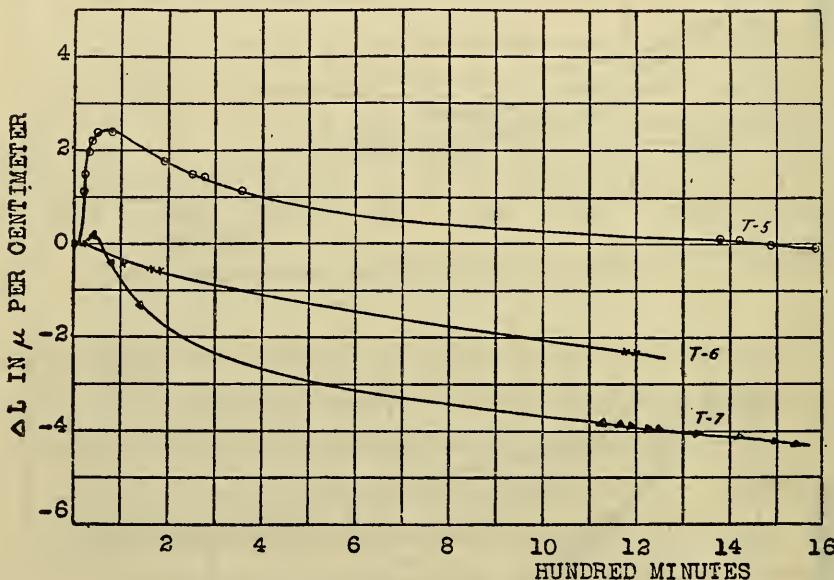


FIG. 20.—Setting changes of synthetic porcelain in water

slightly. Upon being put into the water it evidently absorbed some water and expanded 2.4 microns during the first few minutes; after that it contracted slowly.

The two samples $T-6$ and $T-7$ were kept moist while being adjusted to fit the apparatus and show a slow uniform contraction.

VI. FLOW UNDER COMPRESSION

The authors have found all amalgams yielding under constant pressure even months after amalgamation. Hence the questions of setting and crushing strength become relative factors. When

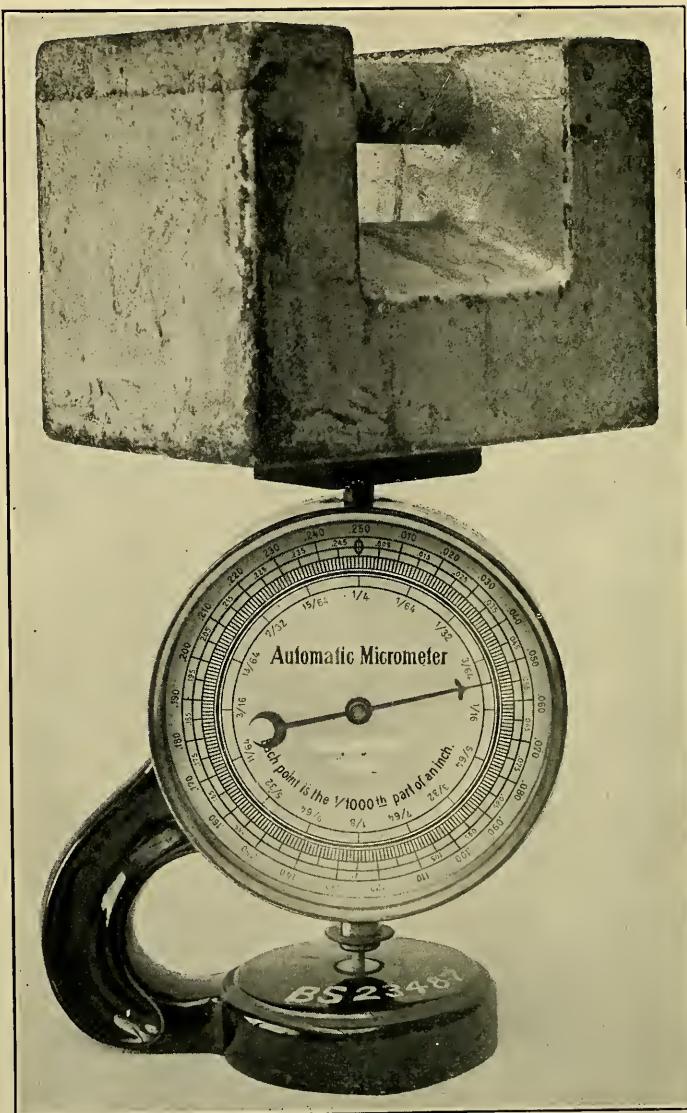


FIG. 21.—Micrometer adapted to measure flow of amalgam

the crushing load is applied as rapidly as possible the crushing strength always runs high.

Most amalgams, after setting 48 hours, will show crushing strength of over 32 000 pounds per square inch if crushed quickly, say in 3 minutes; but by applying a constant pressure of 3200 pounds, only one-tenth the previous crushing load, we found it possible to crush some of these same amalgams within 20 hours.

With these facts established it was decided to make comparative flow tests, beginning 2 hours after packing, and applying the approximate one-tenth load, namely, 3200 pounds per square inch.

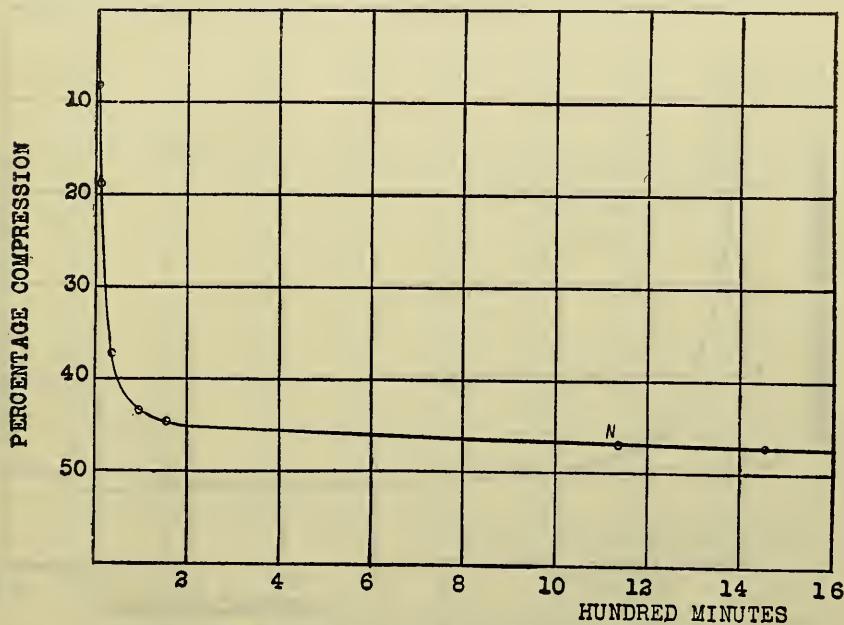


FIG. 22.—Flow of amalgam; load applied two hours after packing

The load was applied to a specimen 4 mm in diameter and about 8 mm long.

The apparatus used for the flow tests is shown in Fig. 21 and consists of a micrometer with a weight pan attached at the top of the upright rod or plunger. The two ends of the specimen are cut at right angles to the axis and placed between the jaws of the micrometer. As the amalgam is compressed by the weight applied at the top of the rod the indicator moves around the dial. The difference of readings gives the amount of compression or flow. An air cushion plunger incorporated in this micrometer

prevents injury to the instrument should the specimens fracture suddenly.

Changes in length are indicated on the dial. These are recorded as per cent change in total length. Figs. 22 to 25 are included to portray the average behavior of amalgams when subjected to this test.

Fig. 22 was plotted from data taken under the above conditions. Two hours after amalgamation and packing the specimen was placed in the compression micrometer and a pressure of 3200 pounds per square inch applied. In 30 minutes it was compressed

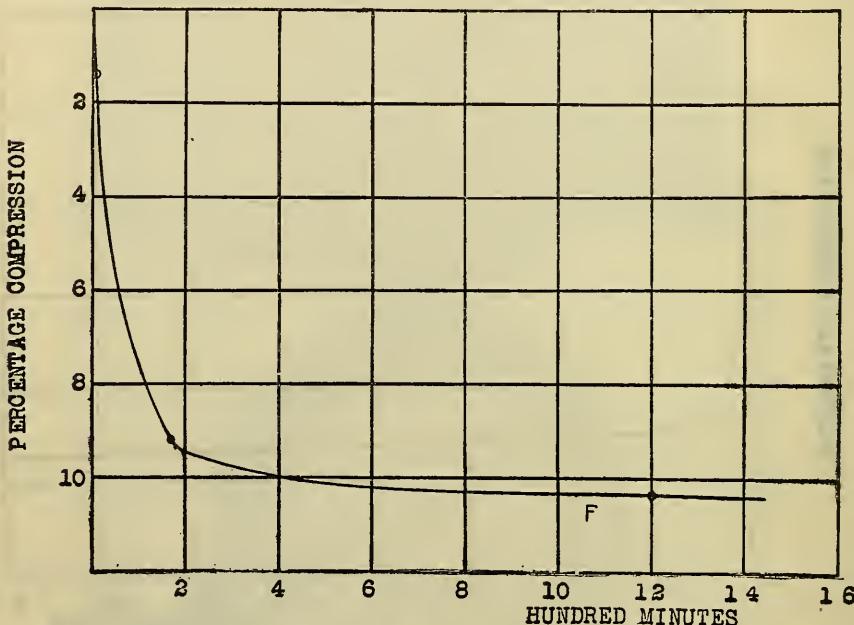


FIG. 23.—Flow of amalgam; load applied two hours after packing

40 per cent. This specimen did not flow materially after 10 hours, due to the fact that the cylinder had been ruptured and was spread over a much larger area, thus reducing the effective pressure per square inch.

There was a possibility of this apparent recovery in ability to resist flow being attributable to delayed crystallization (slow setting), so additional specimens were prepared and retained 48 hours before being subjected to the flow test. The same amalgam under this treatment was compressed 18 per cent and fractured after three days. (See Fig. 25N.)

Fig. 23 is plotted for another amalgam, compression started 2 hours after packing. This alloy was marked "Quick setting."

Fig. 24 (top) indicates the flow of a 5 per cent Zn amalgam and the curve at the bottom is for a nonzinc amalgam. The amalgams in Fig. 25 were not subjected to the one-tenth load until 48 hours after condensing. Amalgam N is the same as that used in Fig. 22.

Most amalgams will withstand this test remarkably well at 1 hour and some at 30 minutes after packing. The qualities producing failure seem to be inherent and permanent in the material (probably chemical compositions) and permit failures after 48 hours as readily as after 30 minutes; the so-called slow-setting or quick-setting qualities being difficult to interpret.

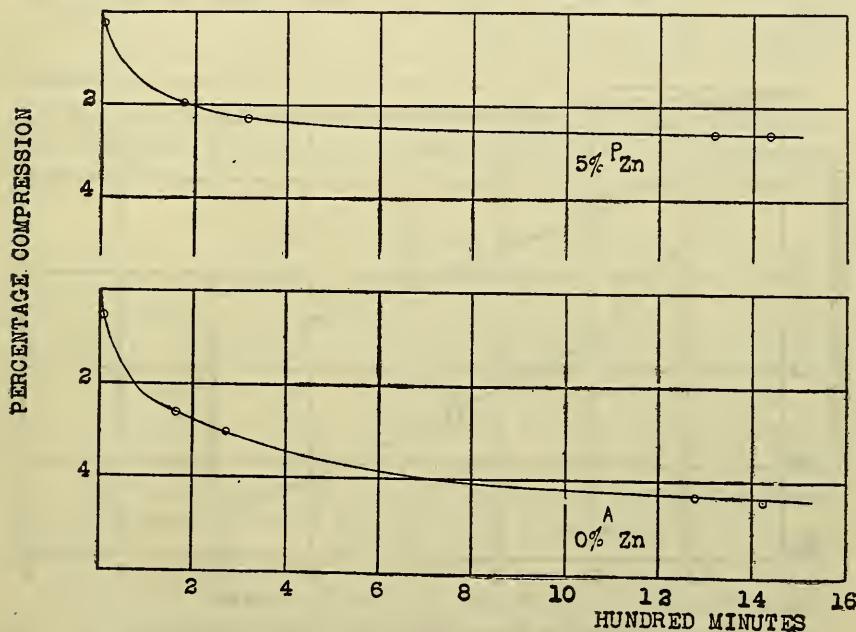


FIG. 24.—Flow of amalgams; load applied two hours after packing

The above may appear to be a new feature, but such is not the case, as it is simply a modification or improvement on the Black dynamometer in which a set of levers operating a dial hand is used to indicate the compression during the crushing tests. The constant, continuous load of one-tenth the crushing value is selected as appropriate for deciding upon the merits of an amalgam when used in a cavity such that it is required to furnish a contact point with an adjacent tooth or filling where constant pressure is applied. The permanence in shape and position of this contact point will eliminate later troubles and constant annoyances due to food par-

ticles which tend to wedge between the teeth. The possibilities of position changes are of importance and the inevitable upsetting of the amalgam tooth margins needs only mentioning to bring one to realize the gravity of such situations. The point of a tooth from an opposite jaw constantly striking an amalgam may apply an effective load of several thousand pounds per square inch even though the gross pressure is only a few pounds. The smallness of the area of contact has the effect of intensifying the impacts when considered in terms of pounds per square inch. The material is being treated in a manner very similar to that employed in battering the head of a rivet where light taps continued for a short time are quite sufficient to change the entire cylindrical end into a flat bur.

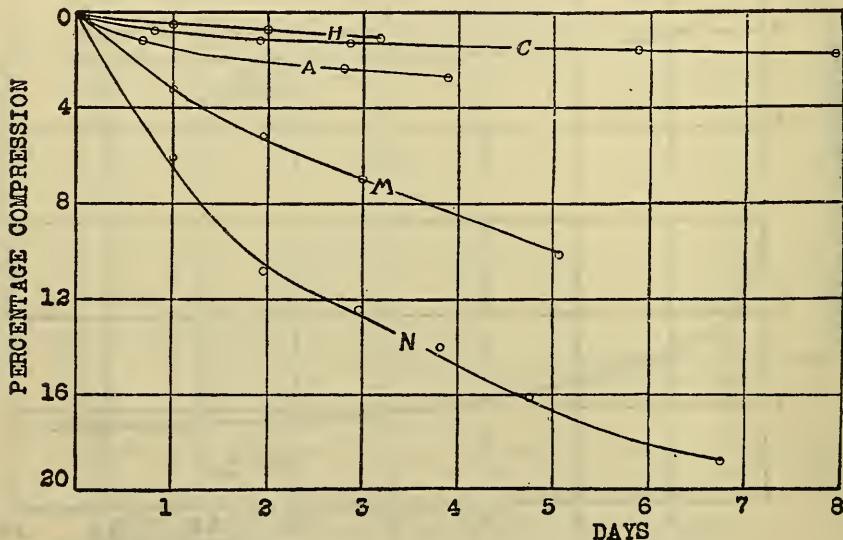


FIG. 25.—Flow of amalgams; load applied 48 hours after packing

VII. CRUSHING STRENGTH

As previously stated crushing strengths have been found dependent upon the time occupied for the test. The tests included in this report were made as rapidly as convenient, using a specimen 6 mm in diameter and 10 to 12 mm long. While not of the size specified by the A. S. T. M. mentioned previously, they are a close approximation to the form. The preparation of specimens 1 inch in diameter and $2\frac{1}{2}$ inches long would introduce factors and difficulties of trituration and condensation which are never encountered in practice, to say nothing of the enormous expense necessary to produce such quantities of alloy.

The regular testing machine equipment of the Bureau was used for these tests. The time required for each crush was about three minutes. Tests were made 14 days after amalgamation. All materials included in this test were made up according to the regular dental technique, in fact, extra specimens were made up by a practicing dentist for comparison and concordant results were found.

By mechanically packing the specimens under high pressure, immediately after amalgamation, it is possible to secure crushing tests of almost twice the values given in the table at the close.

The results of compressional tests are somewhat irregular, but no more so than those found by other observers. It is felt that the comparisons between alloys makes these data worth presenting.

Since these tests were for comparison and were made under similar conditions no extra precautions were taken to control temperature, which was constant at about 25° C, probably within 2°.

VIII. BLACKENING OF HAND

This test consisted in mulling specimens in the hand two minutes after having first been amalgamated for one minute in the mortar. The hands were first washed, rinsed several times, and then dried to make sure that tests were comparable. A zero value indicates that no blackening was detectable; 10 represents a complete and dense black coat or film left in the palm. The values given are the average of several tests by different manipulators. In no case was there a variation of over two points, due to different tests or observers.

The authors will not attempt to pass on the merits of this test. The discoloration of the hand does not a priori imply a discoloration of the tooth tubuli. The free sulphides or combinations of foreign materials producing this blackening require further study to determine their true effect in amalgam usages.

IX. CHEMICAL COMPOSITION

The chemical compositions vary from 45 to 69 per cent silver and from 0 to 5 per cent zinc, copper and tin making up the remainder.

For practically all alloys the claim of balance according to the G. V. Black standard is made, which can probably be interpreted to mean that the resultant filling should expand slightly, never contract, and after a few hours "lay still."

While absolutely exact percentages should produce exactly the same alloys if similarly treated, it is very probable that other factors enter which are far more significant; for example, a certain alloy triturated 3 minutes gave 5 microns expansion in 24 hours. This same alloy gave 15 microns contraction when triturated for 30 minutes. A similar behavior was found for other alloys. Overannealing while exposed to the air at 120°C for 2 hours in an electric oven produced little change in this chemical expansion or contraction, although it did render the alloy slow setting and of granular consistency while being mulled. Balancing an alloy then becomes meaningless unless proper instructions are given for time of trituration, etc.

In this investigation comparatively little interest was paid to the manufacture of alloys since the purpose of the research was to test the finished product. No effort was made to run a set of definite percentages of materials to recommend the proper proportions for a perfect alloy—the companies doubtless prefer to have this left as their field. The properties of cleanliness, size of cut, time of setting, working qualities, annealing, and the like must all be incorporated in the material and each manufacturer will endeavor to strike an average such that the alloy will contain a maximum of desirable qualities.

Certain desirable or undesirable properties are supposed to accompany excessive or deficient amounts of the constituent metals, for example, alloys of low percentages of silver are supposed to be deficient in strength, of poor working qualities, and slow setting; that is, tend to flow badly. These facts were verified qualitatively only. (See Table 3.)

High percentages of copper are said to produce discoloration—this of course should be or may have been settled by the dental profession. However, an amalgam made from an alloy containing 16 per cent copper was discolored when exposed to a weak solution of iodine, very much more than any of four others containing a lower amount of copper.

Zinc has long been considered a "disturbing" element. The claim is often made that zinc is "inadmissible," even in the smallest amounts, and that all amalgams containing zinc will "move." Since this movement is supposed to be cumulative and to extend over a period of years, it is impossible to furnish data extending over a period sufficiently large to be of decisive value.

A number of specimens have been prepared and are under observation. These have been divided into two groups, one of which is kept at room temperature, the other is subjected to temperature variations between zero and 50° C. At the end of the fourth month the first group had changed in length by values ranging from 0 to 0.2 per cent, one zinc amalgam showing the zero change, another zinc showing the 0.2 per cent change. The values obtained by averaging the changes of zincs against the nonzincs, disregarding signs, indicate a more nearly permanent state for the latter.

The group subjected to temperature variations (0 to 50° C) has suffered much greater changes. Here the average departures from initial length appear to favor the zinc amalgams, the average departure being 0.1 per cent, while the average departure for nonzinc amalgams is 0.2 per cent. The extremes of the former are 0.02 and 0.5 per cent and of the latter 0.06 and 0.4 per cent.

Manifestly no definite conclusions can be drawn from the comparison at this time. At a later date it will be possible to give more conclusive evidence bearing on this phase of the problem.

X. ELECTRODE-POTENTIAL DETERMINATIONS

The normal calomel half cell and potentiometer was used in measuring the potentials. A description of this instrument may be found in any text book of physical chemistry. All values except the last are for the resultant amalgam using the alloys specified. A number of amalgams, ranging in values from 0 to 5 per cent zinc, were tested. These are given in Table 2. The potentials are expressed in terms of volts, electromotive force.

TABLE 2

Alloy	Emf	Alloy	Emf
0 per cent Zn.....	-0.54	1 per cent Zn.....	-0.52
0 per cent Zn (duplicate).....	- .55	2 per cent Zn.....	- .52
0 per cent Zn, 16 per cent Cu.....	- .52	5 per cent Zn.....	- .51
0 per cent Zn, 16 per cent Cu (duplicate).....	- .52	Gold (metallic).....	+ .002

No conclusive inferences are to be drawn from these although there seems to be little evidence of excessive contact emf effects due to the larger zinc content.

These tests seem to point toward a complete solution of the zinc (at least) by the mercury. This finding is in harmony with

the practice of giving zinc electrodes a surface amalgamation to prevent local action, due to impurities when placed in the solution of the electric cell.

TABLE 3.—Comparison of Amalgams

Alloy		Crushing strength in pounds per square inch	Per cent flow in 24 hours	Relative blackening	Setting changes, first 24 hours (all values in microns per centimeter)			Claims
Mark	Partial composition				Maximum contraction	Maximum expansion	Final state	
A	Ag 68.... Zn 0....	49 500 51 550	4.4	7	1.5	5.6	+ 4.3 + 4.5 + 2.2	A correct material certified as to balance
B	Ag 67.... Zn 2....	46 500 40 300	3.1	1	3.9	1.5	+ .5 - 1.7 - .2	It is balanced, produces a white amalgam of great strength, free from slightest contraction, has a slight initial expansion never exceeding 3/10 000 inch, takes a high polish
C	Ag 68.... Zn 1....	39 800 38 200 44 200	4.0	0	.6	6.7	+ 5.2 + 6.7 + 4.9	Made in accordance with the investigations of Dr. G. V. Black; is a balanced alloy. This is a perfect alloy in every respect, is age proof, an exact alloy, highest crushing strength, is the only chemically clean alloy made
D	Ag 66.... Zn (1)....	39 950 35 550	3.4	0	.8	5.5	+ 2.1 + 2.6 + 3.7	
E	Ag 60.... Zn ()....	41 950 38 900	9.1	4	9.2	.0	- 6.8 - 5.7 - 5.0	White alloy
F	Ag 66.... Zn 1....	49 500 48 400	10.4+	2	27.1	.0	- 27.1 - 21.3 - 20.2	A balanced alloy made after the approved formula of G. V. Black, M. D., D. D. S.
G	Ag 68.... Zn $\frac{1}{2}$	47 250 46 650	5.0	4	3.5	1.7	+ .4 - .3 - 1.4	Never changes color, contraction nil, expansion 1/20 000
H	Ag 67.... Zn 1....	40 750 40 750	3.7	1	4.1	4.3	+ 3.8 + 3.5 + .7	Used by the U. S. Navy made after the Black formula
I	Ag 68.... Zn $\frac{3}{4}$	38 700 37 100	4.3	1	7.7	.0	- 7.5 - 6.5	Does not shrink, about 1/20 000 expansion takes place; a perfect, nonleaking amalgam can be the only result
J	Ag 67.... Zn 1....	42 750 44 800	7.8	8	1.2	3.8	+ 2.5 + 2.4 + 2.5	A balanced alloy, permanent whiteness, made by the Black method, contraction nil, expansion 1/20 000
K	Ag 54.... Zn 0....	41 550 48 450	9.6	8	2.4	4.9	+ 1.4 + 3.9 + 4.2	Depended upon not to shrink or change form; insures against stained teeth and gutters around fillings
L	Ag 54.... Zn 0....	33 600 38 700	5.3	4	2.1	3.2	+ 2.6 + 3.0 + 2.1	Fillings do not shrink nor change form; has a fine white color which endures; can not shrink
M	Ag 54.... Zn $\frac{1}{2}$	30 400 34 300 30 300	36.0	10	39.1	.0	- 28.1	No shrinkage, no discoloration, a tooth saver; any higher price can not buy a better alloy; an honest, efficient filling material; takes a high polish and keeps it
N	Ag 45.... Zn 2....	30 850 38 500 30 000	47.1	7	7.8	.0	- 3.8	Nonshrinkage, nonexpansion, edge strength, retention of color
O	Ag 67.... Zn 1 $\frac{3}{4}$	40 750 34 000	2.5	2	1.9	.0	- 1.9	
P	Ag 60.... Zn 5....	41 200	2.6	1	7.2	.0	- 4.5	

Table 3 gives a brief comparison of the more important qualities investigated. Some of these are readily verifiable by the interested dentist who is willing to spend the money necessary to purchase the alloys. Others require special apparatus or the services of a testing laboratory, for example, crushing strength and flow, while others require apparatus of such high precision that in addition to the apparatus it is essential to have the tests carried out by a person skilled in the use of such apparatus, for example, accurate determinations of expansion and contraction.

The claims tabulated are those made on the label of the package or in advertisements placed before the public in recent years.

It is a regrettable situation that individual practitioners will find it practically impossible to make complete tests on the materials supplied as balanced alloys to conform to methods of Dr. Black, or any formula or specification. However, it is felt that the manufacturers turning out products which are of a questionable value will welcome any move to place this work on a scientific basis, thus eliminating the necessity for lower standards often used in the fields where price is put before permanence.

There may possibly exist a place for the materials of questionable qualities—that is, tendency to flow excessively or to contract on setting—which necessitates their production and place in the market. If so, specific information should accompany each package, giving full details.

XI. SUMMARY

A survey of the previous work on the physical properties of dental materials reveals a large amount of qualitative work on certain properties with instruments, the inherent errors or sensitiveness of which are comparable with the magnitude of the effect under investigation.

Many of the essential properties have not been considered and in some cases a careless interpretation of results has led to considerable confusion.

Recent work by Dr. Gray, of Milford, Del., with improved apparatus, has indicated the possibilities and importance of obtaining qualitative results on more of the properties. At the request of a branch of the Government the authors undertook such an investigation.

An inspection of the instruments in general use disclosed their lack of sensitivity and necessitated the construction and employment of more suitable apparatus.

Accuracy and efficiency recommended the use of the interferometer for determinations of thermal expansion and setting changes, since the necessary accuracy can be secured with the use of small specimens, the temperature of which is readily controlled.

For crushing strength determinations the calibrated testing machines of the Bureau were used. For the flow tests it was found desirable to select a special instrument. This instrument consists essentially of a precision dial micrometer equipped for applying constant, continuous pressures to specimens.

The electrode-potential measurements were made with the calomel half cell and potentiometer, which is standard apparatus for such measurements.

Careful chemical analyses were made to determine the constituents of the different alloys.

The results of the determinations of the properties tested, which are represented in the accompanying curves and tables, show the behavior of different alloys and the effects of different conditions and manipulations upon the same alloy.

Because of our lack of sufficient clinical experience we have not attempted to speculate on our data except under definite physical conditions, but have attempted to emphasize to the profession some of the important properties, together with a means of determining the same.

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WASHINGTON, November 24, 1919.



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